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Accelerated Life Methods for Understanding Refrigerant Chemical Stability in HVACR Systems

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A Numerical Accelerated Life Methodology for Understanding Refrigerant Chemical Stability in HVACR Systems

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ABSTRACT

Today's air conditioning product and application designers, as a result of climate change contribution concerns from high global warming potential (GWP) refrigerants, are being asked to consider lower GWP refrigerants which may have the potential for decreased chemical stability. Chlorofluorocarbons (CFCs) and hydrochlorofluorocarbons (HCFCs) were developed after years of research and field experiences to determine if they possessed acceptable chemical stability. They presented some chemical stability concerns with materials, however they demonstrated good product reliability when mitigation actions were taken to resolve chemical stability issues. Hydrofluorocarbons (HFCs) were found in general to be more chemically stable than the CFCs and HCFCs. In recent years, the heating ventilation air-conditioning and refrigeration (HVACR) industry has been actively investigating new lower GWP refrigerants, many of which are new molecules with little knowledge available on their chemical stability.

In this paper, we will summarize the current accelerated test methodologies to qualify a new refrigerant for use. The authors will propose a new interpretative numerical methodology for extrapolating small scale highly accelerated laboratory results to real life HVACR operational conditions using a simple thermodynamic model and climate zone data. R-22 will be used as the baseline refrigerant system given the long history of published chemical stability data.

1. INTRODUCTION

HVACR equipment life expectancies are typically 15 to 25 years depending on the application. The authors are aware of water cooled chiller equipment operating in excess of 50 years. Chemical stability of the refrigerant containing systems needs to be designed to be stable enough without being replaced over the above time periods. The ability to assess the chemical stability of refrigerants in the laboratory has been a dilemma in industry for decades. Investigators are challenged to correlate tens of thousands of system operation hours to data from laboratory scale tests performed over durations of days and weeks. Highly accelerated laboratory testing is required because of the impracticality and cost of running equipment for years. It is common practice in the industry to assess chemical stability of refrigerants using sealed glass tubes per ASHRAE Standard 97 procedures with various system materials at highly accelerated temperatures instead of running equipment (ASHRAE 97, 2007). Typical accelerated conditions for sealed glass tubes experiments can vary with temperatures ranging from 130°C to 200°C for durations of 1 to 4 weeks, with a more common test point established at 175°C for 2 weeks. Such temperatures often exceed the inherent thermal capabilities of many organic materials and results from these evaluations may not be relevant to the performance in HVACR equipment.

2. UNDERSTANDING REACTION KINETICS & ARRHENIUS THEORY

Chemical reaction kinetics is the study of chemical reactions with respect to reactions rates and mechanisms. These chemical reactions can be broken down into sequences of one or more single step processes. These reactions can involve reactive collisions between two molecules, or single molecules going through dissociation (degradation) or

isomerization reactions to name a few. These unimolecular reactions (dissociation or isomerization) are often considered first order reactions that follow the general reactions shown in equation 1 and 2. Refrigerant system chemistry chemical reaction kinetics have been studied and the primary chemical reaction usually behaves in this manner with a single molecule undergoing a reduction reaction or a disassociation reaction with itself.

$$\text{Reactant [A]} \rightarrow \text{Product [B]} + \text{Product [C]}, \text{ dissociation} \quad (1)$$

$$\text{Reactant [A]} \rightarrow \text{Product [B]}, \text{ isomerization} \quad (2)$$

The chemical reaction rate, the rate at which these chemical processes proceed, is dependent on numerous factors. Factors that affect the reaction rate include the nature of the reactant, its physical state (solid/liquid/gas), concentration of various reactants or products, temperature, pressure, and the presence of catalysts which may accelerate or alter the reaction mechanism. The rate at which these reactions occur can be described as the change in concentration of the reactant [A] with respect to time (t). For first order reactions, this rate can be described by the first order rate law in equation 3.

$$[A]_t = [A]_0 e^{-kt} \text{ which can be rearranged as: } \ln\frac{[A]_t}{[A]_0} = -kt \quad (3)$$

Rate constants are determined experimentally, and require either; the ability to measure the concentration of the reactant or, knowledge of the chemical reaction and stoichiometry to relate the measured concentration of the products to the depletion of the reactant. If these considerations are not taken into account, the rate study will be ineffective and could lead to confounding results or incorrect conclusions. A typical approach used in evaluating refrigerant stability is to monitor the concentration of a key degradation product (or products) that results from the reactivity of the refrigerant at an elevated temperature for a prescribed period of time.

The knowledge and fundamental understanding of a chemical's reactivity can be further expanded upon through the relationship described by Arrhenius theory (Brown, et al., 2003). Arrhenius argued that for reactants to transform into products, they must first acquire a minimum amount of energy, through heat or other means, called activation energy (E_a). The concept of activation energy is fundamental when describing the sensitivity of reaction rates to temperatures. As the rate constant (k) is utilized to describe the reaction rate at a specific temperature, the Arrhenius theory allows an understanding of the rate constant with respect to temperature (equation 4). Rate constants can be measured over a range of temperatures to develop an Arrhenius expression to describe the fluid's reactivity based on the activation energy, E_a, in kilojoules per mole and the pre-exponential factor A, in 1/sec.

$$k = Ae^{-E_a/RT} \quad (4)$$

Arrhenius theory is fundamental when utilizing accelerated life tests. A typical approach to accelerated testing involves use of elevated temperatures to drive measureable reactions at reasonable time durations. This approach to testing is based on a generalization supported by the Arrhenius theory, in that for many common chemical reactions near room temperature, the reaction rate doubles for every 10°C increase in temperature. This relationship of reaction rate as a function of temperature can then allow a researcher to gain important information on chemical reactivity on a scale of days as opposed to years. For instance, if the chemical system being studied is a system operating at 100°C for 1 year (365 days), by using the generalization from Arrhenius, an accelerated test could be run at 150°C for 11.4 days to achieve similar chemical reactivity, if reaction mechanisms are not altered by the elevated temperature. Increasing the test temperature by 50ºC results in an increased reaction rate per Arrhenius theory by 32 times (2^5), which is equivalent to doubling of the reaction rate for every 10°C rise in temperature (365 days/32 equals 11.4 days). When the appropriate system is chosen and further studied, valuable information can be gained on the reaction kinetics using the Arrhenius relationship. Knowing the chemical reaction kinetics and degradation species allows one to be able to understand the variables which may promote or inhibit reactions. With this knowledge, strategies can be further developed and implemented to achieve successful applications of HVACR fluids.

3. BEHAVIOR OF HVACR REFRIGERANT STABILITY TO ARRHENIUS

The concept of using rate kinetics to study HVACR refrigerants is not novel. However with the introduction of HFCs such as R-32, R-125, R-143a, R-134a, the use of rate kinetics has been infrequent or unnecessary, as these refrigerants are largely chemically stable at highly accelerated temperature conditions, compared to the refrigerants they replaced, like R-22, R-12, and R-115. Many historic papers studied the reactivity and degradation of these past refrigerants in the presence of lubricant and various metals to determine their suitability for use. These fluids, while more reactive, were successfully applied in the industry for more than 50 years. In these early fundamental studies, the chemical stability of the refrigerant and lubricant combinations were evaluated using the sealed tube test approach, a method that is widely applied today to understanding HVACR system chemistry. In these sealed glass tube evaluations, the materials to be tested – typically refrigerants, lubricants, and metals – are sealed in a glass tube, and aged in a heated oven for a specified amount of time to accelerate the aging process. After exposure, the contents in the tube would be analyzed to detect breakdown products for determination of rates of reactivity or decomposition mechanisms. The glass tube approach was employed due to the relative chemical inertness of glass, but more importantly, glass allows the ability to monitor the condition of the materials before, during and after testing. Other approaches to accelerated testing have been applied over the decades, including use of stainless steel pressure vessels (Doerr, et al., 1993) and DSC techniques (Kauffman, 1993), however the sealed glass tube approach largely remains the preferred approach in the industry.

As introduced earlier, one must first fully understand the reactions at hand to ensure the system being studied and measured is appropriate to model the reaction kinetics. In the case of R-12, gas chromatography analysis was utilized to identify and further study its reduction reaction to form R-22 via the exchange of one of its chlorine atoms for a hydrogen atom from the lubricant (Doderer, Spauschus 1966). In this study, direct correlation of R-12 decomposition could be inferred by the measurement of R-22 produced, due to the stoichiometric relationship of the reactants and products in the reduction reaction. In an extension of the R-12 work, the mechanisms of R-22 decomposition were comprehensively studied and discussed (Doderer, Spauschus 1964). From this study, two degradation species were detected, and it was determined that R-22 decomposed both by reduction and disproportionation reaction mechanisms. These studies not only identified the degradation mechanisms of the refrigerants, but they also demonstrated the fundamental difference between the thermal stabilities of R-12 and R-22 at elevated temperatures.

An approach to relating accelerated stability data to the operating temperatures of refrigeration compressors was proposed by the 1964 study of R-22 by Doderer and Spauschus. The approach involved exposing multiple sealed tubes for specific times and temperatures to measure the reaction rates. It was further recognized by the authors that while the decomposition of refrigerants is a complex process, the use of a pseudo first-order rate expression could enable the data to be modeled on a semi-log plot of temperature versus percent decomposition over time (Figure 1). Evaluation of this relationship gave insight that the rate of R-22 decomposition increased by a factor of 1.52 for each 10°C rise in temperature with the presence of lubricants. Using the relationship in Figure 1, the experimental data could be applied to a hypothetical compressor model, which enabled the authors to propose an estimated amount of refrigerant breakdown one could expect over a 10-year life of a compressor. Figure 2 is summary of the same data but applied in an Arrhenius relationship format. While understanding the behavior of reactivity was critical to understanding system chemistry, the industry also needed an approach to readily understand fluid compatibility on a meaningful time scale.

In many of the fundamental rate studies discussed earlier, test durations ranged from days to years. In 1958, the ASHRAE contaminants committee laid out a standard sealed glass tube approach to enable short term tests to compare acceptability of oils for use with fluorocarbon refrigeration systems, specifically R-12 (Kvalnes, 1965). A standard temperature of 175°C was established, with evaluation of the contents occurring at 8 and 16 days. Selection of the temperature and time was based primarily on the ability to generate measureable difference in the interactions of R-12 with lubricant to enable comparative assessments to establish a standard baseline. While this approach was well accepted for R-12 refrigerant system comparisons, the better chemical stability presented by R-22 made it difficult for researchers to have a comparable approach at the same time and temperatures. Researchers decided to keep similar time periods but increase temperatures to drive comparable reactivity. Huttenlocher performed sealed glass tube evaluations at two temperatures and three aging periods to validate the use of elevated test temperatures, using the Arrhenius relationship (Huttenlocher, 1972). In this study, it was recognized that a short coming in continually increasing temperature approach is the potential for driving chemical reactions that would not be typically seen in a

HVACR systems. Instances of "runaway" reactions had been noted in prior research, as well as the concerns of the formation and concentration of refrigerant degradation products leading to increased reactivity uncharacteristic of the refrigeration systems being studied (Sandvordenker, 1985). Huttenlocher's R-22 study at highly accelerated temperature conditions up to 200°C was able to predict R-22 reactivity observed at lower temperatures.

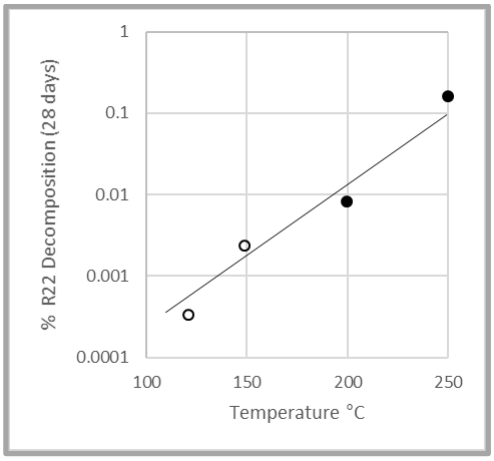

Figure 1. Effect of temperature on R-22 decomposition (Spauschus and Doderer, 1964)

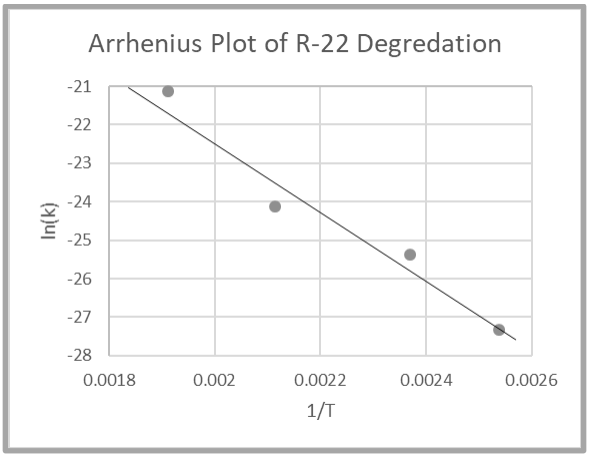

Figure 2: Arrhenius representation of R-22 decomposition (Spauschus and Doderer, 1964)

The application of Arrhenius relationships to the accelerated life testing of refrigerants is not limited to R-12 and R-22 refrigerant systems. In 1990, the authors company in cooperation with a refrigerant supplier, performed a similar sealed tube study with R-123 and paraffinic oils at various temperature and times to propose the total refrigerant breakdown over the product's lifetime. Using the Arrhenius approach, a test temperature and time of 131°C for 11.8 days was determined to equate to 27 years of chiller operation in cooling mode. This example serves to show that a test temperature below 175°C can be used to qualify and characterize operating system reactivity for a fluid successfully utilized in the industry for several decades.

Limited references have been found that make attempts to tie accelerated small scale laboratory testing to real system application life expectancy. Using a methodology of comparing sealed glass tube experiments to field chiller run time is costly, requires a long time to validate, and requires the installation of field equipment. Additionally the sealed glass tube approach, while well established, is not a perfect tool to compare to the chemical dynamics seen in a refrigerant cycle. This approach provides many benefits to the assessment of system chemistry, so long as the analyst accept the system being studied is static and does not perfectly replicate the dynamic conditions present within a compressor or system. In addition, the potential for multi-material interactions and/or low temperature operation are not accounted for which could be significant to overall system impacts. The sealed glass tube method has provided the HVACR industry a tool to better understanding system reactivity without having to test equipment over long periods, but this method requires calibration to refined operating system model to full enable its effectiveness to prevent the investment in costly system tests.

4. EQUIPMENT OPERATION IMPACTS ON REFRIGERANT STABILITY

Investigators have the ability to model HVACR equipment performance to determine the operating conditions, e.g. temperatures, pressures, concentrations and flow rates of the refrigerant and the lubricant. Figure 3 shows a typical temperature operational map for an air cooled chiller. This figure only shows the application temperature conditions, and more insight is needed to provide an accurate description of the refrigerant conditions within the equipment. The actual refrigerant temperatures will be much higher or lower given the approach temperatures in the heat exchangers as well as the isentropic efficiency of the compressor at those conditions. Heat exchanger approach temperatures are typically 5°C to 10°C higher/lower than the required application delivery temperatures. As mentioned earlier, the compressors efficiency and the compression ratio in combination with the refrigerant properties will have impact on the refrigerant temperatures further. Figure 4 shows the effect on compression ratio (P) on compressor discharge temperature (CDT) with R-22 as the refrigerant. Using simple thermodynamic modeling in combination with equipment operational temperatures and compressor performance at those conditions can give good insight on the

maximum temperatures the refrigerant might see in the equipment, and more importantly the duration the refrigerant will spend at various temperatures.

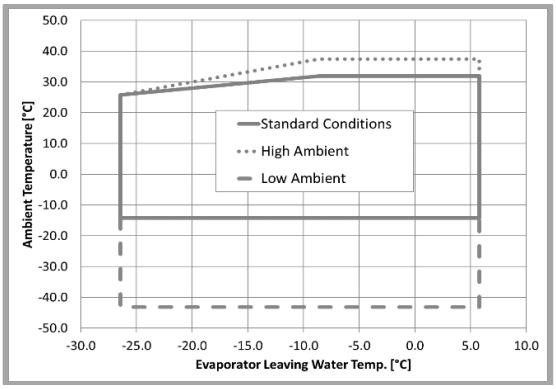

Figure 3: Operating Temperature Map for a Typical Air Cooled Chiller

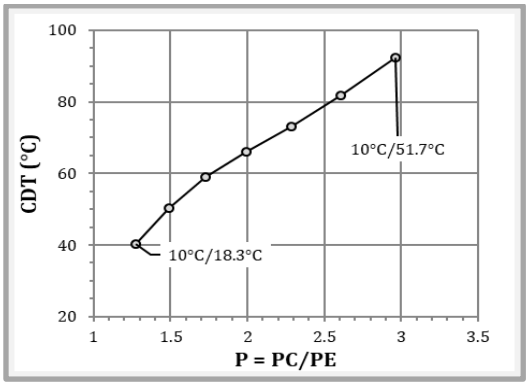

Figure 4: Relationship of Compressor Compression Ratio and Compressor Discharge Temperature

Over the decades as equipment designs and minimum product efficiency standards have progressed, the impact on reducing system temperatures has been significant. In a 1968 paper by Spauschus and Doderer, a hypothetical compressor model was proposed to provide a better relationship to chemical stability data. The refrigerant temperatures used to describe the model compressor ranged from 160°C to 220°C, which are equipment temperatures not typically seen in today's more energy efficient equipment. Increased system efficiency has been gained by reducing heat exchanger approach temperatures and increased isentropic compressor efficiency to name a few. As an example, let's compare the application of R-22 in a piece of unitary rooftop equipment at an estimated energy efficiency in 1968 before minimum product efficiency standards and at today's minimum product efficiency levels. A simple thermodynamic model was used and the refrigerant saturation conditions in combination with compressor efficiency were adjusted to reduce the COP to a level that was typical in the past. The heat exchanger approaches were increased by 5ºC on both the evaporator and condenser in combination with reducing the compressor isentropic efficiency to 50 percent. Operational conditions used were 10°C evaporator/40.6°C condenser/11.1°C superheat/8.3°C subcooling/70 percent isentropic compressor efficiency as the baseline case for today's equipment operating conditions. The resulting COP was determined to be 5.77 using today's conditions. When applying the increased heat exchanger approaches and reduced compressor efficiency, the resulting COP was reduced to 2.92. The impact on refrigerant temperatures resulted in increases of compressor discharge temperature of 32.3°C. This simple demonstration shows that equipment efficiency and application conditions are needed to calibrate any accelerated laboratory tests.

HVACR equipment is a dynamic chemical pump with the refrigerant seeing a wide range of temperatures and refrigerant concentrations, e.g. liquid and vapor continually. Confounding the problem, the chemical stability of the refrigerant is also a function of exposure to catalysts, for example lubricant and metals, at those times and temperatures. Chemical stability of a refrigerant is more than just a function of temperature, but it is a function of the time the refrigerant spends at that temperature with various materials. One could conservatively assume from an accelerated life standpoint that if a unit is operated for one hour that the all the refrigerant is exposed continuously to the high temperature in the unit, e.g. the compressor discharge temperature. However, that would overstate the amount of time the refrigerant spends at that temperature. Analogously, one could assume the refrigerant spends an equal amount of that hour within each component and their temperature conditions, however this still over estimates the time the refrigerant spends at the highest temperatures. Knowing the time spent at various temperatures is needed to further calibrate a laboratory accelerated test methodology.

Determining the time mass distribution of refrigerant at the various system temperatures is a complex problem. One must know various equipment operation variables, for example; refrigerant charge, refrigerant flow rate, volume of system components and amount of refrigerant liquid and vapor in these components. A unitary rooftop system (10.6 kw size) was used as an example to show the distribution of refrigerant and time spent at temperature within system components. First, the total internal volume of the system (43.5 liters) was determined as well as the volume of the individual components. The amount of liquid versus gas in various components was fixed, with the evaporator

containing 30 percent liquid/70 percent vapor and the condenser containing 70 percent liquid/30 percent vapor by weight respectively. The condenser having a majority of the refrigerant liquid is not unexpected since most systems operate with condenser subcooling which drives a need for significant storage of liquid refrigerant.

Given the amount of the liquid and vapor contained in the components and the refrigerant mass flow rate, one can determine the refrigerant's time at temperature distribution. The operational conditions presented previously can be further applied to a simple thermodynamic model to determine refrigerant mass flow rate, pressures, temperatures, and density of the refrigerants at various state points. The refrigerant charge was set based on the cataloged refrigerant charge (3.1 kilograms). Next, using the refrigerant mass flow rate (20.3 kg/hour·kilowatt), and refrigerant volumes in components (proprietary), the amount of time the refrigerant spends per hour was determined at the respective component's temperatures. Table 1 provides a summary of the time and temperature distribution for this operating point. It should additionally be noted that the ratio of time spent in each component will not vary much by the operating condition. The fraction of an operating hour the refrigerant spends in each component will be further applied in later discussions to calibrate laboratory scale accelerated life test to real world operation by climate zone.

Table 1: Time Spent by Refrigerant in Various Component Over One Hour

Component	Temperature	Fraction of Hour Refrigerant Spends at Condition	Refrigerant Time at Conditions (Minutes)
Evaporator	10.0ºC	0.316	19.0
Compressor	32.2ºC	0.090	5.4
Compressor Discharge and Piping	75.6ºC	0.003	0.2 (12 seconds)
Condenser	40.6ºC	0.591	35.4

5. A NUMERICAL APPROACH TO RELATE ACCELERATED TEST METHODS TO HVACR EQUIPMENT OPERATIONAL CHARACTERISTICS

Today, published climate zone data is available through resources like ASHRAE standard 169. It is a simple operation to then use this data in combination with a simple thermodynamic refrigerant model and set the condition of the system to obtain equipment operational characteristics. The climatic data (dry bulb temperature vs hours spent) was obtained for eighteen climate zones that represent most of the globe's climates (ASHRAE 169-2016). Climate data was collected for 2.8°C dry bulb temperature discretized bins from -33°C to 45°C for all climate zones. Climate zones include weather regions from very cold (7), moist zones (6A-0A), dry zones (6B-0B) and marine zones (5C-3C). Figure 5 provides a summary of all the climate hourly data by the discreet 2.8°C dry bulb temperature ranges.

Air conditioning is only needed at elevated ambients, starting at temperatures 18.3°C to 23.9°C, depending on the climate zones humidity conditions. It is then easy to understand the amount of time and temperature the equipment will see over its lifetime. Long equipment operation in relatively low ambients climate zones is not of particular interest from a refrigerant chemical stability standpoint since the equipment's operating temperatures are low. Climate zones that require long hours of operation at moderate to high ambients can set the worst case from a refrigerant stability stand point because it will drive up the condenser and compressor discharge temperatures.

The simple thermodynamic model was run at the 2.8°C dry bulb temperature discretized bins to obtain the refrigerant temperatures for the condenser and compressor discharge temperatures. An approach temperature of 8.3°C was used for both heat exchangers and a compressor isentropic efficiency model was used to account for changes in pressure lift requirements. The evaporator and compressor suction temperatures were kept constant over operating conditions. This is reasonable given the low temperature of these components and resulting chemical reaction rates.

From the component analysis in Table 1, it can be noted that the refrigerant is primarily exposed to lower temperatures and only spends 0.3% (12 seconds every hour) at the high compressor discharge temperatures. Since refrigerant stability is a function of time at temperature, the total refrigerant breakdown for each climate zone could be calculated as a summation using the reaction rates at each component's temperature along with the time spent at each condition over the life of the equipment. A simplified example, before accounting for climatic impacts, would be if a piece of equipment operates for 1000 hours per year with an equipment life expectancy of 10 years, then then the refrigerant would see 3160 hours, 900 hours, 30 hours, 5910 hours at the evaporator, compressor, compressor discharge and

condenser temperature conditions respectively. Next, using chemical reaction rates determined in highly accelerated laboratory experiments, an estimate of the total amount of refrigerant breakdown can be calculated for the above conditions. By further expanding this relationship across the equipment operation map for specific climate zones, a more complete description can be provided of the system, as the equipment will see higher and lower ambient conditions over its life of operation for various operating hours.

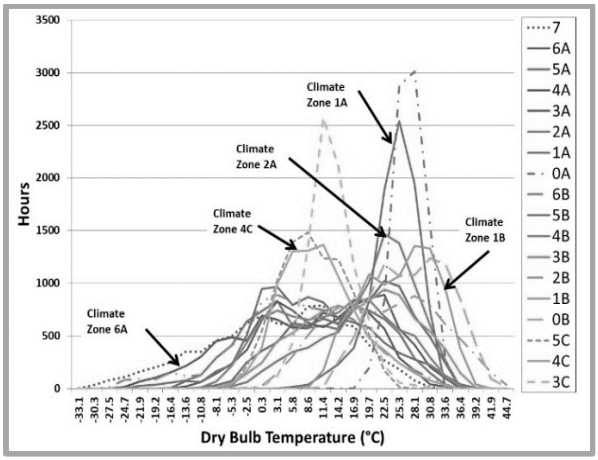

Figure 5: Number of hours spent at various ambient temperatures (bins of 3.4ºC) by climate zone

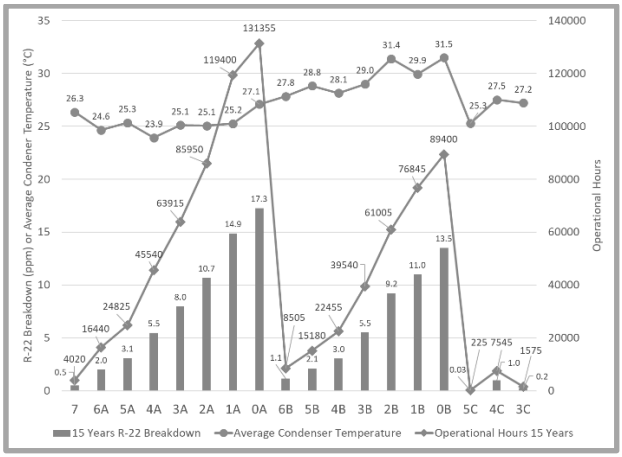

Figure 6: Summary of estimated R22 breakdown by climate zone, over a 15 year life.

From the analysis of these many contributing factors – reaction kinetics, system component analysis, and operation characteristics specific to the various climate zones – trends in refrigerant degradation can be modeled and applied to estimate potential system impacts. Continuing our use of R-22 as an example refrigerant, Figure 6 is a summary of applying the model to see differences in R-32 generation across the climate zones as well as distribution of degradation effects within the system components. From this model, it is evident that aggressive climates drive elevated potential for refrigerant degradation. Equipment operation in climate zone 0A was determined to have the most R-22 breakdown. Further evaluation of the component contributions in this climate zone do not attribute all the modeled decomposition to be due to compressor discharge. Given the modeled reaction rates and elevated residence time in the condenser, 68% of the total refrigerant decomposition is predicted to arise from this component.

The furthered application and validation of this tool can have additional benefits to the analysis of system reactivity, as it can enable better understanding of the relationship (and limitation) of accelerated sealed glass testing. As discussed in prior sections, the industry commonly uses a sealed glass tube accelerated test point of 175°C for 2 weeks. This time and temperature was arbitrarily chosen to accelerate system reactions to a reasonable time span. It is not an approach suitable to evaluating suitability of new refrigerants for use in equipment. When the theory of Arrhenius is used to relate this test temperature to system operations at aggressive climate zones, the accelerating factor used is very large, and likely drives reactivity atypical to the dynamic refrigerant system. Assessment of the system modeled using the 0A climate data indicates an average system temperature of 22°C. In using an accelerated test at 150°C (22°C + 150°C ≅ 175°C) above this average temperature, it can be shown using the Arrhenius theory, that the reactivity is being driven at a rate ~32,000x (2^{15}) greater than the reaction rates that would be observed at the average system temperature. This acceleration is equivalent to 1227 years of equipment operation (14 days times 32,000 divided by 365 days per year), which maybe an unreasonable acceleration factor.

Alternatively, if you choose another system temperature more representative of the maximum compressor discharge temperature modeled with the 0A climate data, we see temperatures closer to 100°C. In relating this operational temperature to the standard testing temperature of 175°C, we now see an acceleration of the reactivity by 181 times ($2^{7.5}$), which would relate back to 7 years of the fluid seeing this temperature constantly (14 days times 181 divided by 365 days per year). As it is understood that the HVAC system is dynamic, with systems seeing a variety of times and temperatures, with <0.4% of its time at the discharge temperature, one can see this 7 years quickly becomes an underestimation of the relation of time to such an accelerate test. With these examples in mind, it becomes evident and crucial that the researcher understands the implications of accelerated testing in choosing appropriate test conditions to develop accelerated test models.

6. CONCLUSIONS

The approach to refrigerant chemical stability assessments has largely remained the same for the past sixty years since it was established in the industry through research on CFC-12 and HCFC-22 by Parmalee, Huttenlocher, Doderer, Spauschus, and Sanvordenker. A new numerical approach is proposed using simple thermodynamic models, equipment design characteristics and climate zone data in combination with laboratory determined chemical reaction rates for a specific refrigerant system for determining the appropriate accelerated temperature and time for relation to equipment operation and life.

NOMENCLATURE

$[A]_t$ – Depleted reactant concentration
E_a – Activation energy (kilojoules/mole)
T – Temperature (Kelvin)
k – Reaction rate constant or coefficient
CDT – Compressor discharge temperature
P – Compressor compression ratio
$[A]_0$ – Initial reactant concentration
R –Gas rate constant, 8.314 joules/mole-kelvin
A - pre-exponential factor for chemical reaction
t – seconds
Pc – Pressure condenser
Pe – Pressure evaporator

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